HISTORICAL PERSPECTIVE ON THE USE OF NIOBIUM MICROALLOYING IN STRUCTURAL STEELS

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Abstract

The first commercial trials of steels with niobium as a microalloy addition occurred in the USA in the late 1950s and were a response to a reduction in the price of niobium following the Korean War together with the discovery of huge reserves of niobium ore in Brazil and Canada. For the first few years, the production of such steels was restricted to the hot strip mill route, but when the mechanism of the effect of niobium on steel was better understood other steel production routes became available. It was soon observed that the properties of structural steels in particular, were significantly improved including strength, toughness and weldability. The first national standard for niobium-treated steels appeared in the UK in 1962. The existence of this standard, together with a better knowledge of the role of niobium in steel, encouraged a world-wide interest and led to a rapidly growing market for niobium-microalloyed structural steels.

Introduction

At the beginning of the 20th century, following the advent of bulk steelmaking techniques, such as the Bessemer and the open-hearth processes, a plain carbon steel was the most commonly used structural material for bridge building, boiler making, shipbuilding etc. The prohibitive cost of high strength alloy steels restricted their use to specialised applications. For example, a steel containing 3.25% nickel, although relatively expensive, was used in the construction of the Queensboro Bridge, New York in 1902. Likewise the Manhattan Bridge in 1906 used a similar steel for the stiffening trusses. The use of these costly steels was justified for structures in which a reduction in the weight or size of members was a necessity [1].

In the 1930s, US Steel developed high strength low alloy (HSLA) steels containing small amounts of copper, nickel and chromium totalling from 0.55 to 2.45%. The presence of the alloys allowed an increase in yield strength from about 250 to 350 MPa. These steels were and still are used for many applications and, although a higher price than carbon-manganese (CMn) steels, they exhibit properties, including atmospheric corrosion resistance that results in economies to the user [1].

Until the 1940s, all the aforementioned steels were typically joined by fasteners such as rivets. However, when welding became more common for joining, then weldability became an issue. At the same time, fracture toughness, often linked to the presence of weld defects, grew in

importance. The time was ripe for the development of microalloy steels with their improved weldability and toughness. Table 1 gives the compositions of some early structural steels together with a modern microalloy steel [2]. The composition of the Sydney Harbour and Melbourne Bridges are typical of their periods, but the exceptionally lean composition of the Forth Rail Bridge was probably only possible as a result of the manufacturing route for plate production at that time in which finish rolling temperatures were relatively low.

Table I. Examples of structural steel compositions over 100 years (19mm, YS 350 MPa).

Structure	C	Si	S	P	Mn	Cr	Al	Nb	CEV
Forth Rail Bridge (1890)	0.23	0.02	0.024	0.046	0.69	*	*	*	0.35
Sydney Harbour Bridge (1929)	0.34	0.20	*	*	1.00	*	*	*	0.51
Melbourne King Street Bridge (1961)	0.23	0.19	0.026	0.017	1.58	0.24	<0.005	*	0.54
Offshore UK(1994)	0.08	0.31	0.002	0.012	1.41	0.027	0.034	0.028	0.32

* no data

The high carbon equivalent value CEV level of the Sydney Harbour Bridge did not play a role in its construction because the assembly was by riveting. However the Melbourne Bridge, which was fusion welded, had major cracking problems and was a good example of what could happen when inexperienced fabricators used a high strength steel (BS 968:1941) which, in some cases, was out of specification with regard to carbon [3]. The table also shows a typical composition for a modern microalloy steel which contains the grain refiners aluminium and niobium and relies mainly on a fine grain size, produced by a normalising or controlled rolling treatment, for its strength and toughness. This approach is in contrast to the older steels which used a higher carbon content to obtain strength and were in the as-hot-rolled condition.

Early Use of Niobium in Steel

Prior to the first successful commercial production of niobium microalloy steel in 1958 niobium had wide use as an alloying element in alloy steels [4]. It was used in the form of ferroniobium to improve the properties of austenitic stainless steel particularly in the USA which started this practice in the mid 1930s. The niobium stabilises the carbide phase and improves the high temperature properties and castability. Niobium was also added to super-alloys based on Fe-Cr-Ni-Co compositions which played a prominent role in the rapid development of gas turbines and jet engines [5].

The fact that niobium was not added to plain carbon steels for about 25 years after it was an addition to alloy steels seems anomalous, particularly since Becket and Franks were granted patents in 1939 – 41 which claimed that niobium within the range 0.02 – 1.00% improved some properties of both alloy and plain carbon steels [6]. This improvement was mainly attributed to grain refinement which raised the tensile strength and gave better impact properties. In several

low alloy steels the niobium content was in the microalloy range. However, the examples presented of as-rolled and normalised plain carbon steels contained 0.28% Nb and gave increases in yield stress of 80MPa and 95MPa for hot rolled and normalised steel samples respectively. Plain carbon steels with lower niobium contents, in the normalised condition, showed much smaller increases in strength. Thus the Becket and Franks patents were important in revealing the ability of small niobium additions to improve the properties of plain carbon steels, but it gave the impression that such high microalloy addition levels were considerably greater than required.

Supply and Price of Niobium

In the 1940s and 50s the largest producer of niobium mineral concentrates was Africa, in particular Nigeria, then a member of the British Commonwealth. Other producing countries were Belgian Congo (Democratic Republic of Congo), Norway, Malaya (Malaysia) and Brazil [5]. The USA was the largest user of niobium minerals and, for example, in 1955 imported around 3.6 million kg of concentrates equivalent to 90% of the total world production [5].

During the Korean War, 1950 - 53, the USA placed restrictions on the use of niobium since it was considered to be of strategic importance and the Defence Procurement Agency paid an incentive bonus of 100% to both foreign and home producers of niobium. USA production was a tiny fraction of the USA total requirement. The incentive bonus started in 1952 and was authorised to continue until December 1958 or until a total of 6.8 million kg had been stockpiled. The resulting price increase of niobium concentrate over the period 1950 to 1958 adversely influenced further development of the use of niobium in the steel industry [5]. However the price of ferro-niobium peaked in the year 1955 and decreased to a much lower level in 1958. Figure 1 shows the price of niobium, in the form of ferro-niobium, in current dollars, i.e. actual historical price, over the period 1940 to 1970 [7]. The relative stability of the price during the 1960s, when supplies from new sources in Brazil and Canada came on stream, greatly aided the acceptance of niobium as an alloying element in steel and led to the widespread and growing use of niobium-microalloyed steels.
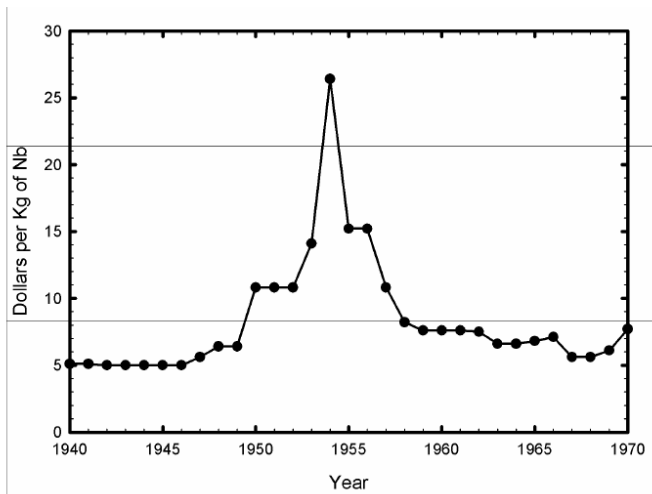
Figure 1. Price per kg contained niobium in ferro-niobium in current dollars (year end average) [7].

Early Trials

In the mid 1950s, huge niobium ore (pyrochlore) deposits were discovered in Brazil (Araxa) and in Canada (Oka) [5]. These discoveries massively increased known world reserves of niobium. In 1957 the Molybdenum Corporation of the USA acquired a 25% share in the mining operation in Brazil (later to become CBMM) [8]. In considering possible new uses for niobium, Molycorp approached some American steel companies with a view to conducting trials to make additions of small amounts of niobium to CMn steels. W.G. Wilson of Molycorp was aware of the work of Becket and Franks and expected that, in the first trial in 1957 at the Homestead Works of US Steel, the as-rolled plates would be grained refined and have improved strength and toughness. The addition used of 0.25 to 1.00kg of niobium (as ferro-niobium) per ton of steel (equivalent to about 0.02 to 0.08% niobium assuming 80% recovery). The heat was made to ingots of semi-killed CMn steel and the ingots rolled to plate using normal schedules. The strength of the plates increased, but they were very brittle [8, 9]. No follow-up trials were carried out due to these disappointing results.

The second commercial trial was conducted at the suggestion of N.F. Tisdale of Molycorp at the Great Lakes Steel Corporation, a division of National Steel of the USA [8]. Again small amounts, 0.11 to 0.45kg, of niobium were added to the ingots of a semi-killed CMn steel [10]. In this trial the ingots were rolled on a hot strip mill at thicknesses up to 13mm and the resultant properties were excellent. Thicker plates up to 38mm tended to have poor toughness due to the presence of large grains in the microstructure, similar to the plates in the US Steel trial. Nevertheless, as a

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result of the very promising results at thicknesses less than 13mm, Great Lakes Steel Corporation started commercial production of the first niobium-treated CMn steels in 1958 [4].

Early Research

The announcement by Great Lakes Steel Corporation that they had entered the market with the GLX-W series of niobium microalloyed steels engendered huge interest among the world's steelmakers. The fascination lay in the fact that such small amounts of niobium, 0.005 to 0.03%, could cause such a large improvement in strength. They stated that the yield strength increased by around 90Mpa and the UTS by 70 MPa which they attributed to a change in the solidification mechanism which produced a fine-grained microstructure [4]. This encouraged other steelmakers to conduct their own trials and to perform research into the mechanisms responsible for the powerful effect of niobium.
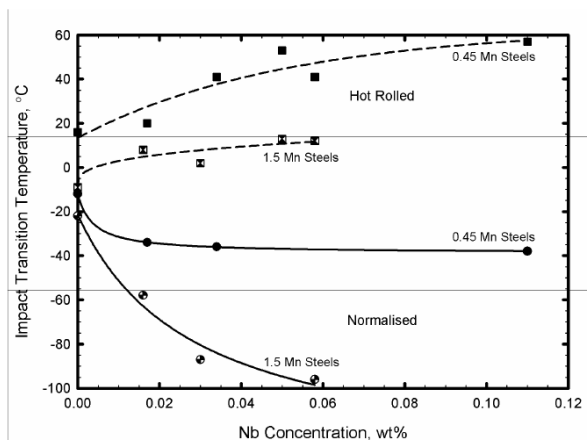

Figure 2. Effect of niobium on the 20 J transition temperature of hot-rolled and normalised laboratory steel [11].

The first full laboratory investigation was carried out by C.A.Beiser of Union Carbide, Niagara Falls, New York, who studied commercial samples from the Great Lakes Steel and steel made in the laboratory. The results were contained in a preprint which was never published [11]. Beiser found that increasing the niobium content of the 15mm as-rolled plates increased strength while impairing the Charpy properties, despite providing some grain refinement. This detrimental effect of niobium on impact properties was totally reversed after a normalising treatment (Fig. 2). Beiser recognised that grain size changes alone could not explain the effect of niobium and he suggested that grain-boundary networks of a carbide phase in the niobium-treated as-rolled samples caused the observed reduction in impact properties. He also associated this brittle network with an increase in yield strength.

A major European steelmaker, Colvilles Ltd of Motherwell, UK, was also involved in trials in 1959 in which niobium was added to CMn structural steel plates. Results similar to those of US Steel were obtained. In thick as-rolled plates (> 12 mm), a small niobium addition increased strength, but reduced impact properties. Colvilles could find no obvious microstructural features which could explain the mechanical property change [12]. W.C. Leslie of US Steel commented that "unless economical means can be devised to eliminate the detrimental effect of columbium on notch-toughness of hot-rolled plate steels, it is likely that columbium-treated steels will be used principally in sheet form, for which impact properties are less important" [13].

Further development of niobium-treated steels, particularly for the thicker plates and sections needed for structural purposes, required the discovery of the mechanisms for the niobium effect. Accordingly in 1959 Colvilles initiated a one year programme of research at Sheffield University, Department of Metallurgy, which was the first to utilise the Petch Relationships [14, 15] in the study of commercial steels:

$$\sigma_y = \sigma_o + k_y d^{-\frac{1}{2}} \tag{1}$$

$$\beta T_c = \ln\beta - \ln C - \ln d^{-\frac{1}{2}} \tag{2}$$

where σ_y is the lower yield stress, σ_o is the lattice friction stress opposing the motion of a dislocation, $k_{y\,is}$ a measure of the localised stress required to propagate general yielding across the grain boundaries and d is the grain diameter. Also, T_c is the impact transition temperature, β is a material constant related to σ_o and C is a measure of the difficulty of propagating a crack.
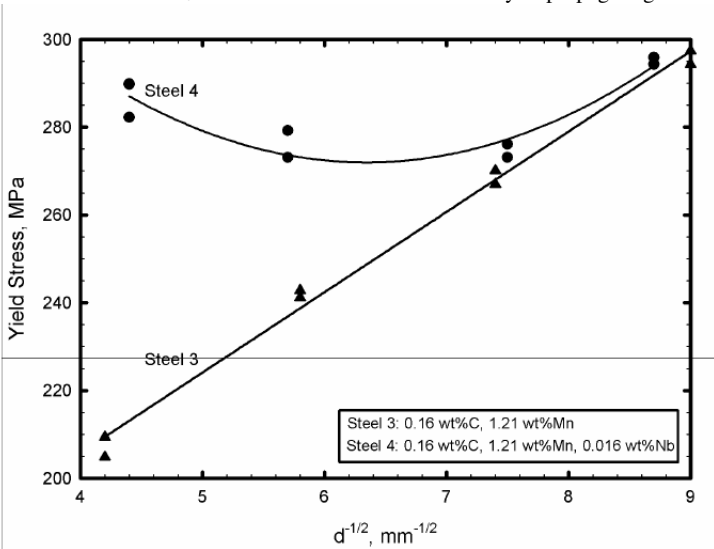

Figure 3. The influence of 0.16% Nb on the relationship between σ_y and $d^{-\frac{1}{2}}$ in a commercial

0.16% C   1.21% Mn steel [16, 17]

These relationships were utilised in an attempt to separate the grain refining effect of niobium from any other effect it may have. Although σ_o and k_y are both constants for a given steel it was found that the niobium-treated steels in the study did not obey equation (1), as shown in Figure 3 [16, 17]. At the coarse grain sizes obtained using high austenitising temperatures, it was clear that niobium had an effect on the yield stress in addition to its effect on grain size. Moreover, niobium raised the impact transition temperature independent of grain size. By utilising the basic data presented by Heslop and Petch [18], it was deduced that niobium caused an increase in σ_o [16]. This was suggested to be a result of the presence of a very fine precipitate of niobium carbide, nitride or carbonitride [16, 17].

In further research by Morrison [19], direct evidence for the presence of fine carbonitride precipitates was presented and it was shown that they were generally in the form of rows, later explained by Gray and Yeo [20] to be due to precipitation on the advancing α/γ interface during transformation. Data from Morrison [19] showing the influence of niobium content on yield stress in hot-rolled steels are presented in Figure 4 in terms of the Ashby-Orowan relationship [21]. This predicts that the strengthening is caused by precipitates mainly within a size range between 5 and 10nm.
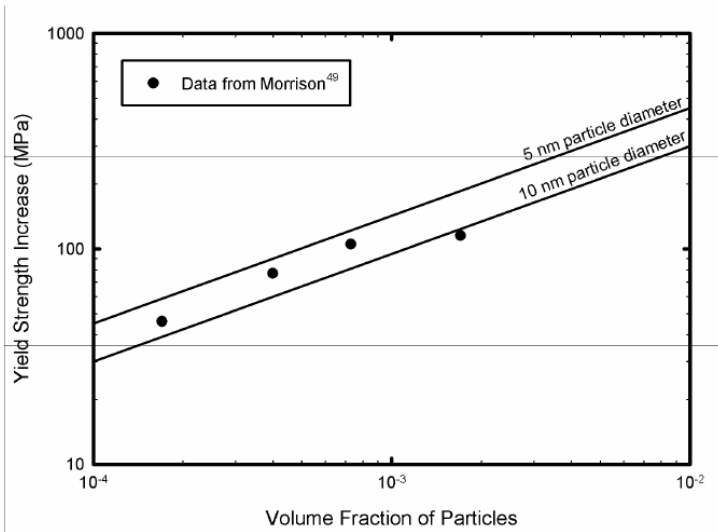

Figure 4   Precipitation strengthening in 0.12% C 0. 50% Mn laboratory steels containing 0.004 to 0.15% Nb [19] with particle diameter calculated from the Ashby-Orowan relationship [21]. The yield strengths have been corrected to a constant grain size of 12µm.

Another important effect of niobium discovered in the early research was that in solution in the austenite, it reduced the transformation temperature into the bainite range particularly at higher manganese levels of around 1.5% [19, 22]. Figure 5 shows CCT diagrams obtained by Ronn [22] and described by Kazinczy et al [23]. The occurrence of a coarse austenite grain size in a plate during hot rolling was found to provide the condition necessary for the formation of an acicular microstructure during the final cooling of the plate. This had the effect of intensifying the embrittlement caused by the presence of a coarse grain size and precipitation hardening.
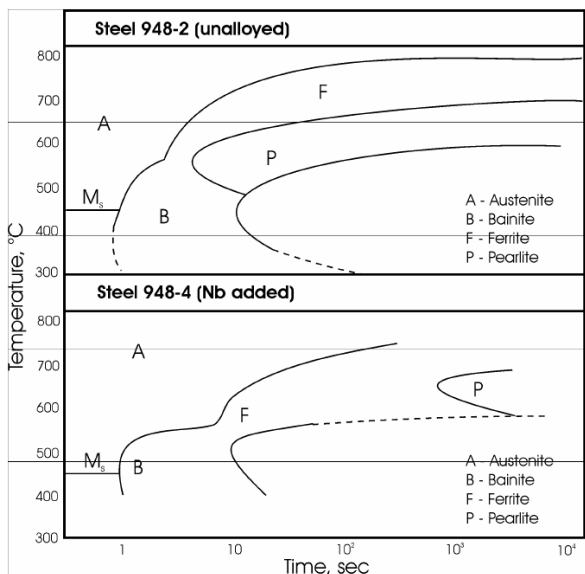

Figure 5. The influence of 0.036% Nb on the transformation characteristics of a 0.11%C 1.01% Mn steel [22, 23].

Improved Processing

Mackenzie [24] observed that finish rolling temperatures below about 900°C gave good notch ductility and naturally occurred in as-rolled plates up to about 13mm thick. Greater thicknesses could be tolerated in structural sections due to their slower rolling rates and consequently lower finishing temperatures (Figure 6). It was obvious that in order to obtain the full benefit of the niobium microalloying in plates, the hot rolling process had to be controlled to the lower finishing temperature and refine the grain size such as occurred in the hot strip mill.

Vanderbeck [25] had observed in 1958 that some European plate mills were practicing controlled low temperature hot rolling aiming at a temperature in the final pass of around 850°C. Plates of CMn steel up to 50mm were being rolled in this way to improve the impact properties. Such rolling practices were not widespread due to the significant resultant loss in production rate.

When similar rolling procedures were applied to niobium microalloyed steels, the results were inconsistent. In the mid 1960s, it was found that niobium greatly reduced the recrystallisation rate of deformed austenite. This property of niobium had to be taken into account in developing rolling schedules, which gave a fine uniform grain size in the final product [26, 27]. Very successful thermo mechanical controlled rolling (TMCR) procedures were eventually developed for niobium-treated steels which gave excellent mechanical properties in plates over a wide thickness range with minimum disruption to output.
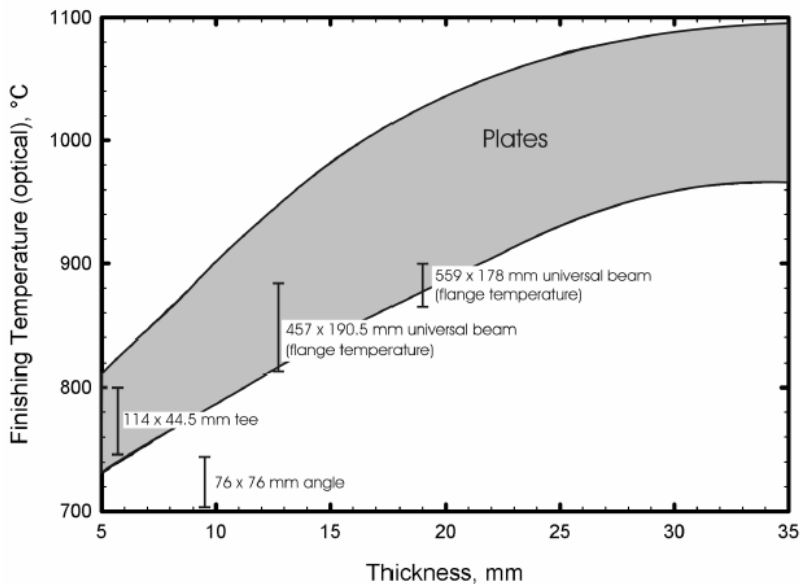

Figure 6. General relationship between thickness and finish rolling temperature for plates and sections [24].

Early Structural Steels

At the time the first commercial niobium microalloyed steels were being produced, the commonly accepted structural material was CMn steel plates and sections. These CMn steels were used as structural members in bridges and buildings and in the welded plate form for the construction of ships and storage vessels and other applications. The main advantages of ordinary CMn steel were its low cost and general ease of fabrication. Although high strength low alloy steels had been available for many years at the time, the high price and more complex fabrication requirements were such that they appealed only to engineers who required a high strength structural steel in specialised circumstances. However, the addition of a small amount of niobium

to the normal, low cost, semi-killed CMn steel was able to transform it to a higher strength steel which was profitable both to the steel producer and the customer. Unlike the low alloy high strength steels which had carbon equivalent values (CEV) significantly greater than those of CMn steels, the addition of niobium had no influence on weldability:

$$CEV = C + \frac{Mn}{6} + \frac{Cr + Mo + V}{5} + \frac{Ni + Cu}{15} \tag{3}$$

Of course the addition of niobium also had the potential to be used to decrease the CEV while maintaining steel strength.

The first niobium-treated CMn plates produced from the hot strip mill at Great Lakes Steel Corporation had a yield strength of up to 415Mpa and were used for small pressure vessels and structural members in trucks as well as for linepipe [10]. It is well recognised that when newly developed steels are incorporated into a national standard they become much more widely accepted and sales increase. The first national standard to involve niobium-treated structural steels in plates and sections up to 50mm thick was introduced in the UK in 1962. Difficulty in welding structural steels with high levels of carbon and manganese (0.30%C, 1.5% Mn) had led to the introduction in 1941 of BS968 which limited the carbon content to 0.23%. The increasing demand for a high strength, weldable steel was met by making a micro addition of niobium to the BS968:1941 steel with lower carbon and manganese contents which allowed an improvement in weldability while maintaining or increasing strength. This BS968:1962 steel compared favourably with an existing ASTM steel (ASTM A441) with vanadium as the alloying addition and the European DIN 17100 St 52 steel, a fully killed aluminium-treated steel. Figure 7 compares the yield stress guarantees for BS968:1962 with those for BS968:1941 for plates and sections. The niobium-treated steels of BS968:1962 had a particular advantage in terms of strength at thicknesses greater than 13mm because of the powerful grain refining affect of niobium in this balanced steel during a normalising heat treatment. It has been pointed out that although suitable for cold forming, hot forming introduced problems for the new standard [24]. Below 13mm the steel, being in the hot-rolled condition, was not suitable for hot forming since the precipitation strengthening of the niobium would be lost. Above 13mm it was required to hot form from the normalising temperature otherwise the grain refining effect of the niobium would be impaired.

Another early use of niobium was as an addition to steel for boilers and other pressure vessels. A comparison of design stresses at elevated temperatures for niobium-treated, aluminium-treated and silicon-killed CMn steels showed that the niobium-treated steel was superior over a range of temperature from room temperature to 400°C [24]. The British Standard was amended in 1964 to allow the inclusion of niobium as a microalloy addition (BS1501: 213, 223) which allowed a higher guaranteed yield stress and a superior notch ductility. Many fabricators used such a steel to build pressure vessels [28].
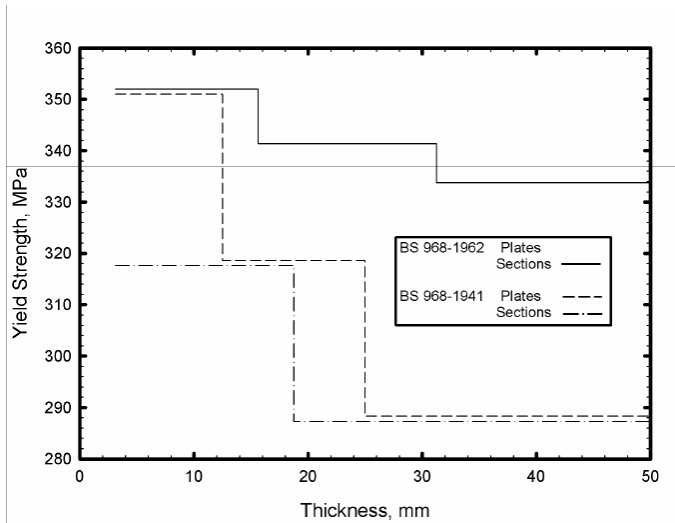
Figure 7. Yield strength guarantees for plates and sections to BS968: 1941 and BS968: 1962 [24].

Discussion

Despite some knowledge of the benefits of making small niobium additions to CMn steel, which existed before 1940, it took until 1957 before a commercial trial was carried out. There are various possible reasons for this. The cost of niobium (as ferro-niobium) was very high in the mid 1950s as a result of stockpiling by the USA (1952 - 58) and this undoubtedly would have been a barrier to its use in CMn steels. However, the cost of niobium was actually lower in the 1940s, than it was throughout the 1950s and 1960s and so price alone could not have been the only reason for the delay in its use as a microalloying element.

There was a trend in the early research to use additions of niobium an order of magnitude greater than microalloying levels[6,29], hardly surprising since even the amounts of vanadium and titanium were typically 0.1 – 0.2% in early steels [2]. Thus, even at the price of niobium which prevailed in the 1940s, such high levels would have made it an uneconomic alloying element since the usefulness of an alloying addition is judged by assessing its effect on properties related to its cost. Also, its reputation was as an addition to stainless steels and super-alloys to provide specialised properties unrelated to those required in structural CMn steels. The required amount of niobium to meet these needs was obtained from mining operations in Africa, but world reserves were limited and certainly insufficient to create a whole new market opportunity for niobium. With the discovery of the very large reserves in Brazil and Canada in the mid 1950s, there was the potential to open new markets. Thus Climax Molybdenum, who had a share in these

reserves, chose to trial niobium as an addition to CMn steels and had the insight to use niobium levels within the microalloying range which would be economically acceptable. An important contributing factor at this time was the reduction in the market price of ferro-niobium towards the end of the stockpiling by the USA (1952-58).

Even as commercial production of thin niobium-treated plates commenced, there was only a rudimentary understanding of the mechanisms whereby niobium influenced steel properties and this slowed further development. However the international community, whose interest was sparked by the fact that such a small, relatively low cost, addition of niobium could produce such a large change in mechanical properties, began a series of research programmes which eventually led to the development of the large family of niobium microalloy steels we have today. This research, which thrived on cooperation between steel companies and universities, was essential for the success attained by microalloy steels. It should also be noted that the success of niobium-microalloy steels encouraged research into the other microalloying elements, vanadium and titanium, which has greatly benefited their use.

It was critical to the commercial acceptance of niobium-treated structural steels to emphasise to the engineer the economic benefits of using such steels which ranged from weight reduction and hence energy savings to lower construction costs. The ability to be able to add niobium to the ordinary semi-killed CMn grades was a considerable advantage in promoting the early niobium-treated steels since it substantially reduced production costs [24]. This was only possible because of the relatively weak deoxidation power of niobium [30]. This advantage no longer applies since later developments have led to the production of fully killed steels of higher quality.

The versatility of niobium as a microalloying element became evident, after the initial CMn steel development, with its addition to steels produced using various manufacturing routes including as-hot-rolled, normalised, controlled rolling and quenching and tempering. A wide variety of steel compositions was involved such as conventional low alloy steels and pearlitic steels with carbon contents less than 0.08% [31]. Such steel developments, in combination with the stable pricing and supply position of ferro-niobium served to maintain the dominant position of niobium as the prime microalloying element in structural steels.

Conclusions

1. Although the beneficial effect of small additions of niobium on the mechanical properties of laboratory-made CMn steels was determined in the late 1930s, it took 20 years before the first commercial trials took place. These trials were initiated due to a combination of factors including the discovery of huge reserves of niobium ore in Brazil and Canada in the mid 1950s and the need to find new markets to utilise this additional supply of niobium. The discovery of the reserves coincided with a reduction in the price of ferro-niobium following the stockpiling in the USA which occurred during and after the Korean War.

2. The relatively high price of niobium compared to conventional alloying elements made it impractical to use as an addition to structural steels at typical alloying levels. However, at the appropriate microalloying levels, the niobium had a sufficiently beneficial influence

on the mechanical properties and weldability making the steel attractive to fabricators who were able to reduce the amount of steel used to compensate for the increased cost. The stability of supply and pricing since that time has been important in maintaining the use of niobium in structural and other steels.

3. A lack of detailed knowledge of how niobium influenced steel properties was an initial barrier to the development of niobium-treated structural steels, but a large international research effort, started soon after the first commercial production began, filled this gap and allowed continued successful development.

4. The wide acceptance of niobium as a microalloying addition to structural steels was due to its versatility in being able to influence such diverse mechanisms as grain refinement, precipitation strengthening, transformation strengthening and austenite recrystallisation which resulted in the development of steels with a large variety of properties and production routes

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